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REVIEW OF SEWAGE AND INDUSTRIAL WASTE LITERATURE-1949. I. STREAM POLLUTION. II. ANALYTICAL METHODS

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Environmental Health Center
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REVIEW OF SEWAGE AND INDUSTRIAL WASTE LITERATURE 1949

I. STREAM POLLUTION

by

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REVIEW OF LITERATURE ON STREAM POLLUTION - 1949

Abatement and Control

Water pollution legislation was adopted in a number of states during 1949. California (7) passed an Act which provided for a 13-man board with nine regional boards. New York and Vermont (74) each passed Acts which enabled the state to join the New England Interstate Water Pollution Control Commission. Michigan (6) broadened its law and increased penalty fines. Instead of the 5-man Stream Control Commission, Michigan now has a 7-man Water Resources Commission, authority being increased to cover groundwaters, flood control and beach erosion. Bloodgood (20) concurs in the trend towards broad powers for state control boards.

Fleming and Ewing (28) outlined the National Water Pollution Program as authorized by Public Law 845, 80th Congress. The Public Health Service with its Water Pollution Control Advisory Board, the Water Pollution Control Division in Washington, the Environmental Health Center in Cincinnati, and 14 river basin offices, is administering the financial and technical assistance to states. State responsibility is recognized. No grants or loans were appropriated. Hart (32) reviewed the Water Pollution Control Act from the standpoint of petroleum refineries and indicated that the industry should examine its position and plan now for necessary treatment plans.

Bloodgood (21) stated "in general, the court evidence gives backing to the law", in reference to stream pollution but indicates a need for a compilation of all available court evidence that shows the

condition of streams declared to be polluted by the court. Murdock (55) indicated that water works legal departments must consider riparian rights, nuisance laws and statutory laws dealing with stream pollution regarding protection of water supplies from pollution.

Watson (73) describes the various types of interstate compacts, commissions and committees for water pollution control as of September 1, 1948. There were four compacts operating with the concurrence of the federal government, one interstate agreement (Incodel), and three additional informal pollution control instruments, involving 21 states and the District of Columbia. The Ohio River Compact (16,73) has authority for the issuance of orders. It requires a minimum of 45 per cent suspended solids removal from domestic sewage and industries can probably be compelled to provide similar primary treatment.

The New England Interstate Water Pollution Control Compact, as described by Scott (61) and Weston (74), increased its participating states of Massachusetts, Connecticut and Rhode Island by adding New York during 1949. This compact operates with enforcement by the individual states, where necessary, not by order of the compact. Streams are classified as A, B, C, or D on the basis of stream use. Many streams in Massachusetts (74) will require complete sewage treatment plants, including effluent chlorination, in order to be improved to Class C.

Allen (4) described the activities of the Interstate Commission on the Delaware River Basin (Incodel) as coordinating existing agencies of state and local governments in New York, New Jersey, Pennsylvania, and Delaware with reference to stream pollution and conservation of the Delaware

River and its tributaries. Philadelphia and Camden have underway construction programs totaling $75,000,000. and a program involving $35,000,000. State and $20,000,000. Federal funds is underway to clean up coal mining wastes on the Schuylkill River in Pennsylvania (10).

The Interstate Sanitation Commission (New York, New Jersey and Connecticut) was described by Hess (34) as being concerned only with the coastal, estuarial and tidal boundary waters. It has established definite chemical, bacteriological, and physical standards for these waters and can issue orders to meet requirements.

The first annual report of the Ohio Valley Water Sanitation Commission (58) has established standards of treatment for the special areas in the vicinity of Cincinnati, Pittsburgh and Huntington-Ashland-Ironton. The Commission also undertook joint studies of oil pollution in the basin and a special study of the Wabash River. Based upon lists of pollution offenders from the eight states, notifications were issued to 1,159 municipalities and 1,422 industries.

LaBosquet (49) describes the treaty of 1909 between the United States and Canada and the surveys of 1913 and 1946 of the St. Clair River. The latter survey was a joint undertaking involving several federal, state, and provincial agencies. Final conclusions of the International Joint Commission have not yet been submitted to the governments of the two countries.

Pond (55) has pointed out the importance of proper conservation and use of water resources. He indicates the necessity for planning by all

interests, the setting of standards for water uses and the development of a realistic and effective program to conserve water. An editorial reports[15] that the sum of $850,000. has been allotted to the states, territories, and District of Columbia for grants to study water pollution resulting from industrial wastes. In addition, $150,000. was allotted to state and interstate agencies for special studies and $1,200,000. to the Public Health Service for operation, research, administration and planning. It was pointed out editorially[13] that sanitary engineers and public health officers have a job of selling to do on clean-stream laws. Another editorial[12] indicates that 42 states have anti-pollution laws and that those vary greatly in the control powers granted.

Winget[77,78], in discussing the relation of industry to stream sanitation, points out: (a) most industrial wastes, such as those from pulp and paper industry, are not a health menance or bacterial pollution problem, (b) individual industrial waste treatment procedures and facilities have to be developed in the laboratory, and (c) industrial waste treatment cannot be financed by public funds but must be paid for with operating funds.

Milligan[52] indicated that the Pennsylvania Sanitary Water Board requires a definite percentage reduction in B.O.D. as the criteria of treatment; 35 per cent for primary treatment and 85 per cent for complete treatment. The Board also plans to establish standards in pounds of B.O.D. pollutant for each of 16 types of industrial wastes. Editorial comments[9,11] indicate that Pennsylvania has obtained cooperation from 47 collieries on the Schuylkill River, 114 of 136 on the Susquehanna River

and six on the Lehigh River in a drive to eliminate anthracite pollution. The program on the Schuylkill will cost $35,000,000., has resulted in 99.9 per cent reduction in solids in the river, and should result in $15,000,000. of salable anthracite fines.

Kittrell (43) and Clark (24) described the stream sanitation program of the T.V.A. indicating that, although not directly concerned with water quality of the streams, the T.V.A. assists state agencies in surveys, planning, educational activities and public relations. Fox (29) states that five industries on the Buffalo River have installed expensive plants for treatment of oil and acid wastes. These industries also agreed to buy Lake Erie water for cooling purposes from the Buffalo Sewer Authority and to discharge spent water into the Buffalo River to supplement low flow periods.

Sulfite waste liquors cause a large part of the total pollutional effect in the Androscoggin River as described by Chase (23). There is an apparent benefit in odor control by the addition of sodium nitrate to the river.

An editorial (14) states that San Francisco has let bids for most of the work necessary to end pollution of San Francisco Bay by domestic sewage. Another editorial (8) states that the Washington State Pollution Control Commission found dangerous contamination by sewage of all Puget Sound and Elliott Bay beaches with 100 sewer outfalls discharging into the Sound.

Hoffert (36) and Kolasinki (45) point out the importance of effecting the removal of industrial wastes from streams, the latter

particularly stressing means of handling waste sulfite liquor. Warrick (72) enumerated the principal industries causing pollution problems in Wisconsin as: canneries, paper mills and dairy establishments and mentioned the progress in waste treatment in those industries.

Kittrell and Thomas (44) explain the legal aspects of the government's liability regarding damage by flooding to existing water supplies in connection with impoundments. T.V.A. was required to furnish a supply equal in quality to the previous supply to any community whose system was actually flooded. Beneficial effects of impoundage enumerated were: more nearly constant stream flows, reductions in coliform concentrations, turbidity, alum requirements, and chlorine demand, reduced fluctuations in characteristics, such as hardness, alkalinity, pH, color and manganese. Adverse effects of storage have been color removal, density currents, structures in navigable channels and tastes and odors.

Woodward and LeBosquet (79) also point out improvements and damages to water quality as a result of reservoir operation. Improvements may result due to: (a) increased dilution of wastes due to increased stream flow, (b) prevention of injurious minimum-flow conditions, and (c) reduction of temperature of streams used for cooling waters. Damages may include: (a) contaminating upstream water supplies by impounding free-flowing tributaries to the reservoir, (b) possible heavy algal growths, and (c) great fluctuations in stream flow if the reservois is operated for power generation. Benefits and damages may be estimated in monetary terms in most cases.

Biology and Bacteriology

Cohn (25) recommends that aquatic biologists and limnologists work with sanitary engineers and chemists to arrive at an adequate understanding of stream conditions and the effects of pollution.

Henderson (33) showed that although the lower Shenandoah River is relatively free of pollution by accepted physical, chemical and bacteriological standards, it contains virtually no fish nor other aquatic animals because of toxicants from industrial wastes. The Surber bottom sampler is a useful device to evaluate the impact of pollution on fish-food organisms and consequently on fish.

The role of the algologist in safeguarding municipal and industrial water supplies was outlined by Taft (66). By predicting algal blooms, algicidal treatment can be initiated before serious difficulties arise.

Johnson (39) discussed the effect of hydrographic and chemical conditions of lakes upon the biology of plankton populations and their vertical and lateral movements. The position and fluctuation of the heaviest concentrations of plankton should be considered in determining levels from which water for domestic use should be drawn.

Abbott (1) found that the rate of production of oxygen by the freely developing mixed cultures of very small algae in sewages containing trade wastes is proportional to the quantity already produced and inversely proportional to the time elapsed.

Moyle (54) reported that algicidal treatment of four Minnesota lakes resulted in an increase in fish production over five adjacent

untreated lakes. Apart from an apparent increased tolerance of Aphanizomenon to the copper sulphate algicide, it appeared that ordinary algae control had little effect on plankton and aquatic seed plants. The toxic effect of copper accumulation in bottom muds is, however, still unknown.

Lackey (48) finds plankton blooms occurring at any time of the year when optimal light, temperature, pH, food (especially inorganic nitrogen and phosphorous) occur. The still inadequate algae control methods fail to completely prevent blooms resulting in acute nuisance conditions. Furfural derived from the methyl-pentosan content of the phytoplankton of some waters was determined in some acidified water samples by Skopintsev (62). The furfural varied from a few to 100 parts per billion in Russian river and lake waters, and up to 600 p.p.b. in swamp waters.

Allen, Blezard and Wheatland (5) found that cyanogen chloride and another unknown substance toxic to rainbow trout, are formed when the cyanides and thiocyanates in gas liquor effluents are treated with chlorine doses smaller than that required to give a residual chlorine.

McKernan, Tartar, and Rollefson (51) after a thorough study attributed the decline of the Olympia oyster industry in lower Puget Sound to sulfite waste liquor pollution (S.W.L.). Oysters subjected for 575 days in 13.0 to 128.9 p.p.m. of S.W.L. were definitely harmed: tolerance lies between zero and 13.0 p.p.m. S.W.L. A modification of the Pearl-Benson test for S.W.L. was presented which consisted in the application of photoelectric colorimetry together with methods of minimizing the effect of the variable water color.

Van Horn [69] pointed out that efficient kraft pulping operations can prevent toxic concentrations of the sodium salts of resin acids and of the blowdown condensates from reaching streams.

Experiments testing the toxicity of D.D.T., benzene hexachloride chlordan and chlorinated phenol insecticides applied as various emulsions and solutions to various Alaskan salmonids, were reported by Cope, Gjullin and Strom [26]. D.D.T. in acetone and fuel-oil solution, chlordan in acetone and fuel oil, and Toxaphene in acetone, were effective blackfly toxicants likely to be safe to fish and fish-food organisms.

Hoffman and Surber [37] found that aerial application of a D.D.T.-oil spray of one pound per acre in two Pennsylvania watersheds killed several fish species in ponds, lakes and streams. Three days after spraying 70 to 80 per cent of the stream insects, including fish-food organisms, were destroyed. Toxicity to amphibians was negligible, but mortality of water snakes was high.

Chang [22] discussed the possible dissemination of cysts of the dysentery ameba in inadequate water supply systems. Ketchum, Carey and Briggs [41] conclude that an organic, heat-labile, antibiotic probably produced by marine bacteria rapidly reduces the number of coliform organisms in domestic sewage disposed into sea waters and estuaries. Further study of this antibiotic and adequate knowledge of principles governing the mixing of water masses are indicated in order to plan efficient marine sewer outfalls.

To produce and market oysters suitable for U. S. Public Health Service certification, the Virginia Health Department [71] regulates all phases of oyster culture, preparation and distribution.

Wilson (76) observed that the microbiotic agents of self purification in streams are found in the same gradients in sewage treatment plants. These organisms are divided functionally into binding, free-living and scouring groups, the successions of which are affected by food supply and dissolved oxygen content. A D.D.T. emulsion, equivalent to 1 p.p.m. of the total daily sewage flow, controls filter flies without the destruction of the biological film. Bartsch and Churchill (18) found no abrupt biological improvement in the North Fork of the Flambeau River near the artificial reaeration site, but improvement in the downstream recovery zone is accelerated. The sulphite pulp effluents have an immediate toxic effect upon most organisms and alter the biological picture for twenty stream miles. In downstream areas the organisms increase, reach a population peak which is followed by a linear decrease as their biochemical activities change the degree and character of pollution.

Chemistry

At a symposium on the oxygen balance (53) Moses gave a general outline of the dissolved oxygen and biochemical oxygen demand tests as applied to streams; McQueen discussed sampling, sample preservation and handling and the subsequent effect these steps have on the reliability of B.O.D. and dissolved oxygen data; Madison advocated the use of an oxygen consumed test in the evaluation of industrial wastes. Kononov (46) noted greater utilization of oxygen by dilutions of Moscow River water than was used by the undiluted river water and concluded that this effect was caused by the presence of a toxic substance in the river water. The study of such effects was recommended as a criteria useful in ascertaining the presence of toxic materials.

Ruchhoft and Ettinger (60) discussed factors bearing on the transportation or destruction of industrial wastes in surface waters, and the subsequent control or accentuation of tastes and odors, due to such materials, by various water treatment processes.

Besozzi and Vaughn (19) discussed the problem of producing a palatable drinking water at Whiting, Indiana. The raw water is polluted by both industrial wastes and domestic sewage. All available water treatments were tried. Activated carbon treatment was the only method which gave them a satisfactory product. Relief from the pollution load due to the sewage is expected.

Stream Standards

There appears to be a marked trend toward stream zoning. Agar (2) cited the details of recent New York legislation which requires stream zoning on the basis of the interests of the public after public hearing. Quality standards are to be fixed on the basis of zoning allocation. The West Virginia Water Commission (75) reported zoning action in the Potomac basin. Criteria for zoning are based on the use of the water for public supply, fish culture or recreation. Weston (74) reports an agreement made in 1946 between the engineers of Massachusetts and Connecticut for classification of their interstate waters on the basis of stream usage. Weston further reports that no streams leave Massachusetts in a nuisance condition although such conditions do exist within the State boundaries.

The Maryland Water Pollution Control Commission has established sewage effluent standards (50). Industrial waste standards are based on the satisfactory ecology of the stream below the discharge. The Policy

Committee recommends that standards be set up considering all factors and all parties involved. King, et al [42] suggest that it would aid industrial control of pollution if a specific industry were allocated a reasonable amount of such stream resources as dissolved oxygen and acid neutralizing power on the basis of study of the specific stream involved. Hoak [35] discussing this paper gave qualified approval to the paper.

Establishment of standards for discharge into the Cincinnati pool by the Ohio River Valley Sanitation Compact was reported by This Journal [17]. Substantially complete removal of settleable solids and 65 per cent reduction of B.O.D. are required.

Strecter [65] reported recent trends in proposed standards of stream sanitation and discussed trends in the further development of standards. Stevenson [63] discussed the evaluation of standards for bathing waters, fish culture, shellfish grounds, boating and camping. Akerlindh [3] discussed factors concerned with the establishment of tolerances for short time discharges of B.O.D. or bacteriological loads. Gorman [30] cited recommendations by Morgan for maximum tolerances of radioactive materials in water.

Oxygen Sag and Self Purification

Thomas [67] presented a method for the application of the oxygen sag curve to stream data, employing nomographs. The sag curve formulation is modified by the introduction of a third constant, "k_3" which takes cognizance of a variety of factors such as deposition or scouring of solids and non-parallel flow. Howland [38] pointed out that Thomas' sag method had much in common with a method of calculation previously

presented by Howland. Thomas [68] agreed that Howland's method had a great deal of merit, but maintained that his formulation [67] was better adapted for application to streams of rapidly changing characteristics.

Streeter [64] presented a nomographic method for solving the oxygen sag equation. Velz [70] considered sludge accumulation, run-off and stream velocity as factors affecting the oxygen sag. Gotaas [31] found that up to 25 per cent of sea water increased rates of biological oxidation. Further increases in the concentration of sea water resulted in decreased rates of oxidation. The sea water concentration did not affect the magnitude of the first stage B.O.D.

Nothlich [57] pointed out factors affecting the oxygen balance of the North River below Hamburg. The domestic sewage of Hamburg is given primary treatment. A dilution factor of 20:1 for waste given primary treatment is considered satisfactory. Oxygen absorption of 4 to 5 g./sq. meter/day is considered a further criteria of satisfactory decomposition.

Kooijmans [47] discussed the pollution and self purification of the River Vecht. This river is especially important because it eventually becomes a part of the water supply of the city of Amsterdam.

Ettinger and Ruchhoft [27] found that the aerobic destruction of phenol and the cresols in natural waters is largely the result of biochemical attack. Factors controlling the rate of attack on such materials are discussed and curves are presented which permit estimate of the low temperature persistence of phenolic materials. Kamp [40] presented a detailed report of damage done in the Potomac basin by pollution resulting from soil erosion.

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REVIEW OF LITERATURE ON ANALYTICAL METHODS

1949

The effect of temperature (5° to 20°C.) and sea water (0 to 18,300 p.p.m. chlorides) on the B.O.D. of sewage in bicarbonate dilution water was reported by Gotaas (1). The results showed that the rate of biological oxidation varies considerably under similar conditions. The date indicate that the rates are higher for low concentrations (550 to 6000 p.p.m. chlorides) of sea water than in fresh water. For higher concentrations of sea water (greater than 9000 p.p.m. Cl) the rates are progressively less than in fresh water. Sea water does not significantly affect the magnitude of the first stage B.O.D. of sewage but retarded the second stage B.O.D.

Comparative studies of dilution waters on the B.O.D. test were reported by K. L. Kick (2). There was little choice between bicarbonate dilution water and mineralized phosphate water with or without supplemental nitrogen for determining the B.O.D. of raw or settled sewage. However, similar to other studies, the fifth and 10th day B.O.D. values were found to be excessively high when river samples were diluted with water fortified with ammonia salts as compared to good agreement between bicarbonate and Formula "C" dilution water B.O.D. values.

From determinations of a three day B.O.D. and a six day B.O.D., Rhame (3) calculated "k" and "L" values on various raw screened, and settled sewages. The "L" values fortuitously approximated the dichromate oxygen consumed test developed by the author. The "k" values varied from 0.071 to 0.413 by the simplified determination which may be subject to major error in the estimation of the "L" or ultimate B.O.D.

The methylene blue relative stability test was placed under automatic continuous observation by Ingols [4]. Bottles (300 ml.) of aerated sewage were dosed with nitrate and methylene blue and placed in a photometer light path. Four photometer sets were connected to a single recording potentiometer and the reduction of methylene blue to the colorless salt was measured at two minute intervals for each sample. The results obtained were considered unsatisfactory.

Abbott [5] reports that the addition of 1.5 to 4.8 p.p.m. of methylene blue to the B.O.D. dilution water suppresses nitrification of NH_3 to nitrite and inhibits the reduction of nitrate to nitrite while permitting the oxidation of organic matter to proceed at a normal rate.

The effect of various environmental factors on the biochemical oxygen demand of activated sludge was reported by Dawson and Jenkins [6]. The results were obtained on 1 to 5 ml. of activated sludge in Warburg or Bancroft respirometers. The respirometers were supported on the frame of a shaking machine and submerged in a water bath. The apparatus was described and graphs showed consistent and reproducible data.

After subjecting over thirty organic compounds to replicate dichromate oxygen consumed tests at two acid concentrations, Moore et al [7] proposed refluxing 50 ml. samples for two hours in the presence of 25 ml. of N/4 dichromate and 75 ml. concentrated H_2SO_4 and back titrating the cool residual oxidant with standard ferrous ammonium sulfate. Ortho phenanthroline ferrous complex was used as an inside indicator having a very sharp endpoint. Some seven compounds of over 30 selected

for study were only slightly oxidized by both the dichromate method and an iodic acid method. Except for glutamic acid which was 86 per cent oxidized by the dichromate method as against 99.9 per cent by the iodic acid method, the dichromate method was more efficient and reproducible in oxidizing those compounds which are amenable to chemical oxidation. The limitations of the dichromate, permanganate, ceric sulfate, and iodic acid methods are discussed. Additional studies on the permanganate oxygen consumed at 30° to 40°C. and at boiling as compared to B.O.D. was reported by Beeckman and Beaujean (8).

An exhaustive investigation of the nitrite test has been made by Rider and Mellon (9). The study showed that superior accuracy and reproducibility could be achieved by diazotizing the sulfanilic acid at a low pH and coupling the diazotized complex with alpha naphthylamine hydrochloride at a higher pH than recommended by Standard Methods (10). Proper pH adjustment may be secured for each step by altering the concentration of acid (HCl) in the two reagents and using sodium acetate buffer in the coupling step. The paper should be consulted for details on the preparation of the reagents, the procedure, and the accuracies which were achieved when using a Beckman DU spectrophotometer.

Baillie (11) used a photometer and alpha-naphthylamine hydrochloride in HCl for the nitrite test according to Standard Methods (10). The hydrochloride solution was easier to prepare and had superior keeping qualities. Good reproducibility was reported when transmittance of standards were compared with a calibration curve. It is questionable, however, that the claimed accuracy of "one per cent or less" was reliable

over the range of 100 to six per cent transmission or 0 to 0.46 p.p.m. nitrite nitrogen.

In photometric analysis the plotting of absorbency against logarithm of concentration has important advantage in evaluating the accuracy [12]. Normally the most reliable range falls between an absorbency range of 40 to 80 per cent.

A modified Sprengel reaction for the determination of nitrate nitrogen was reported by Provedi [13]. Ten ml. of water sample treated with sodium acid salicylate were evaporated on a steam bath. Concentrated H_2SO_4 was added. After warming for several minutes on a steam bath distilled water and a caustic solution were added and the nitrate determined colorimetrically.

According to Sotter et al [14] the suspended solids of raw and settled sewage and activated sludge effluents at a particular plant were estimated with an accuracy comparable to the Gooch method. Four hundred to 800 ml. samples were homogenized in a Waring blender prior to measuring the absorbency in a photometer. A 15 ml. cuvette having a 20 mm. light path was filled with blended sample and placed 145 to 165 mm. distant from the photo-cell to magnify the scattering of light due to turbidity. Depending on the type of samples a range of 20 to 100 p.p.m. suspended solids fell in the reliable photometer range for a 20 mm. light path.

A test for settleable solids was suggested by Ludwig [15] from a study of the average results of suspended solids in raw sewage. Ten sewages were settled under quiescent conditions in 200 ml. beakers, and the supernatant suspended solids were determined every 15 to 30 minutes for four hours. The same sewages were also coagulated without chemicals

for 15 minutes and settled for 20 minutes before testing the supernatant suspended solids. From an analysis of the settling curves it was deduced that the ultimate removal of suspended solids by plain subsidence corresponded to the removal achieved from mixing the raw sewage for 15 minutes at a paddle speed of 72 r.p.m. followed by 20 minutes of settling. This relationship suggested the use of the latter technique in determining the ultimate volume of settleable solids as a quick substitute for the 2 hour Imhoff test.

The relatively inferior assay for volatile acids in sewage sludges by Standard Methods [10] was greatly improved by H. Heukelekian and A. J. Kaplovsky [16]. The proposed assay consists of (a) clarifying the sludge adjusted to a pH of 3.5 with acid, adding ferric chloride, filter aid and filtering; (b) concentrating the filtrate by adjusting the pH to 11, evaporating and cooling; (c) adjusting the pH to 4.0, saturating the residual solution with $MgSO_4$ and steam distilling the volatile acids, and (d) titrating the distillate with standard NaOH to pH 8.2. Recoveries of 92 to 98 per cent of acetic, proprionic and butyric acid additions to sludges were reported as contrasted with inferior recoveries by Standard and other modified procedures.

Kirschman and Pomeroy [17] define the "oil" content of oil-field waste waters as the nonsaponifiable hexane-soluble fraction which is relatively nonvolatile at normal laboratory temperatures. Comparative tests by two modified techniques gave similar results. One technique consisted of extracting the oil from 2.5 liters of water with three 200 ml. portions of chloroform, distilling off the $CHCl_3$, treating the extract with ether-alcohol and caustic, extracting the nonsaponifiable matter with hexane, evaporating the hexane, and weighing the residue.

The alternate technique consisted of flocculating a 2.5 liter sample with 5 millimols of zinc acetate and sodium carbonate, filtering and air-drying the settled floc, washing the original bottle with hexane, extracting the "oil" from the floc in a Soxhlet with hexane and, after evaporation of solvent, weighing the extract. Flocculation with ferric hydroxide appeared less suitable than aluminum hydroxide or zinc carbonate on waters containing appreciable sulfide. Zinc carbonate was preferred since floc filtered more readily than $Al(OH)_3$.

A procedure for the grease content of sewage sludges having minor variations over conventional procedures was reported by Stephenson (18). The acidified sludge (pH 2) was mixed to a smooth paste with an equal weight of $MgSO_4.H_2O$. The mixture was dried, ground, and extracted for 5 hours with petroleum ether. The extract was then dried and weighed.

A methylene blue method for determining hydrogen sulfide was reported by Fogo and Popowsky (19). Hydrogen sulfide was absorbed from a stream of gas in a suspension formed by adding sodium hydroxide to a solution of zinc acetate. The suspension then containing the sulfide as zinc sulfide was treated with an acid solution of p-aminodimethylaniline, followed by the addition of a small amount of ferric chloride solution. After time had been allowed for the formation of the methylene blue, the solution was diluted in a volumetric flask and an aliquot was transferred to a photometer for measurement of the methylene blue color at a wave length of 650 millimicrons.

The addition of malonic acid as a sequestering agent and gum arabic as a stabilizing agent permitted the photocolorimetric estimation of

copper in surface waters by the color reaction with dithio-oxamide [20].

The dithizone method for the determination of copper was modified for use on sewage and industrial wastes by H. G. Swope et al [21]. After destroying the organic matter by wet combustion with nitric and sulfuric acids, the copper was complexed with dithizone at controlled pH and extracted with CCl_4. The optical density of the color complex was measured in a photometer using a green filter (510 millimicron wave length) and referred to a calibration chart. The method was recommended for copper in amounts 0.04 to 14 p.p.m. The standard deviation of the method on 12 to 15 samples of sewage containing 0.2 to 0.3 p.p.m. copper was 0.02 to 0.03 p.p.m.

Strandskov, Marks and Horchler [22] have shown that the residual the residual chlorine test on sewage and sewage effluents by the phenylarsenoxide-amperometric titration method correlates well with the bactericidal results as compared to the poor correlation if the ortho-tolidine residual was used. The method consisted of adding a known excess of phenylarsenoxide followed by KI. As I was liberated by residual chlorine it reacted with phenylarsenoxide. The pH was then reduced to 2.5 with HCl and the unreacted phenylarsenoxide was titrated with standard iodine to an amperometric endpoint.

Palin [23] differentiated free chlorine, monochloramine, dichloramine and trichloramine from each other. Free chlorine or free chlorine plus 2/3 of trichloramine was obtained by titrating a sample containing neutral ortho tolidine and sodium hexametaphosphate with standard ferrous solution until the blue color was discharged. Then KI

was added, and the titration continued to determine monochloramine. To the same sample dilute H_2SO_4, followed by $NaHCO_3$ was added and the titration continued to determine dichloramine. Trichloramine was determined on a separate sample by extracting with chloroform.

Mahan [24] assembled an amperometer from stock items and a few special parts for use in determining the endpoint of residual chlorine titration. A sample of water was titrated with a standard solution of arsenite. The results of three readings (one having an excess arsenite and two deficient in arsenite) are plotted to locate the endpoint for free residual chlorine. The total residual was determined by adding KI to the sample before titrating with arsenite as in the case of free chlorine residual. In the presence of substance having appreciable iodine demand at low pH the total residual may be in some error.

According to Trakhtman [25] active chlorine must be determined at the natural pH of the sample without acidification since adjustment of pH changes the active Cl content.

A review of the most important advance in methods of water analysis during the past five years was made by S. K. Love [26]. The review covered tests for Al, Fe, Ca, Mg, Na, K, Cu, Zn, Pb, Mn, NH_4, B, Se, SiO_2, SO_4, PO_4, Cl, F, CO_2, CN, CNS, NO_2, NO_3, Br, alkalinity, hardness residual chlorine, D.O., B.O.D., dissolved solids, organic substances, turbidity and odor.

The determination of iodine, dissolved oxygen and "active chlorine" based on the photometric estimation of iodine in a solvent extract was

reported by Kulenok [27]. The iodine was extracted from water with several portions of CCl_4 or $CHCl_3$. Errors of 1 to 5 per cent were reported in the 1 to 0.2 p.p.m. range, respectively. The D.O. was determined by extracting the iodine liberated in the Winkler procedure. Errors within 0.3 per cent were reported for the 8 to 12 p.p.m. range. The "active chlorine" is converted to I by addition of KI to samples buffered at pH 4.5 before solvent extraction. In the absence of oxidizing agents other than "active chlorine" solvent extraction of acid solutions was possible with less control of pH.

The procedure for the field test for D.O. was as follows: (a) fill 100 ml. g-s bottle with usual precautions, (b) add required drops of $MnSO_4$ and Alk-KI-Azide, shake and allow precipitate to settle, (c) use a rubber bulb on pipette to quickly withdraw 50 ml. of clear liquor followed by quickly adding the required acid and 10 or 20 ml. of CCl_4, shake or (c-c) add 20 ml. CCl_4 to inside of bottle allowing clear liquor to overflow followed by the required drops of H_2SO_4 to acidify. Shake, and (d) bring samples to laboratory to determine iodine in extractant photometrically. (If bottles are French Squares that have been previously checked for the optical density with 20 ml. of CCl_4, it may not be necessary to remove the extractant from the bottle). The accuracy was reported within 10 per cent in the range 1 to 10 p.p.m.

A colorimetric method for minute concentrations of D.O. [28] was compared to the potentiometric method developed by Porley. The modified Winkler method uses Alk-$MnCl_2$, HCl and ortho tolidine to yield the

yellow color of chlorinated ortho tolidine. The method was somewhat more reproducible than the potentiometric test in the lower levels of the range 0.08 to 0.003 p.p.m.

R. W. Martin [29] reports the use of 4-aminoantipyrine as a reagent for the colorimetric estimation of phenol in water and salt brines in the range 0.2 to 2 p.p.m.

A new indicator for the determination of carbonate alkalinity was proposed [30]. The indicator was di-Na 4,4-bis (1-amino-1-naphthylazo)-2,2' - stilbenedisulfonate. Its preparation was described and, as it hydrolyzes readily in water, it was made up in acetone. Since it was susceptible to chlorine, one drop of 0.1 N. $Na_2S_2O_3$ was added. The color change was from pink to blue.

A comparative study of the total and magnesium hardness of water by the soap method and the Schwarzenbach titration (Versenate) method was made by Rossum and Villarruz [31]. When the technique of finding the soap endpoint on borate buffered samples was modified from Standard procedure [10] and an empirical formula of hardness equivalence was used the results were satisfactory and in agreement with the Versenate Schwarzenbach titration [32]. The latter method consisted of titrating a sample with a solution of versenate (the disodium salt of ethylene-diaminetetra-acetic acid) in the presence of an indicator (Eriochrome-Swartz T) and an ammonium chloride-ammonia buffer. The indicator changes sharply from pink to blue in daylight and from pink to almost colorless in artificial light. The method requires minor modification for samples containing 0.1 p.p.m. or more of manganous manganese. Magnesium hardness

may be determined after the calcium has been precipitated as the oxalate. The authors conclude that it seems desirable to use the Versenate method for total hardness and the soap method for calcium.

A simple procedure based on the well known reaction with titan yellow was described for the visual colorimetric determination of magnesium in water (33).

Control tests on the determination of calcium using the principles of Chan's urea hydrolysis technique were made by Ingols and Murray (34). Photomicrographs and results showed that the calcium more readily precipitates in large crystals when a sample acidified to pH 1.0 was treated with ammonium oxalate and urea, and then boiled.

Studies on the newer lithia-silica types of glass electrodes for pH meters showed a very rapid response to wide changes of pH in buffered solutions (35). The pitfalls of inaccurate measurements due to improper washing of electrode, mixing the solution, and allowing the electrode to dry were evaluated and discussed.

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